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Optimization of headspace sampling using solid-phase microextraction for volatile components in tobacco

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Abstract

Solid-phase microextraction (SPME) was evaluated as a tool for headspace sampling of tobacco samples. Several experimental parameters (e.g. sampling temperature, pH, moisture, and the type of SPME fibers) were optimized to improve sampling efficiency in two aspects; maximum adsorption and selective adsorption of volatile components onto SPME fibers. The effect of these parameters was often dominated by the physical and chemical nature (e.g. volatility, polarity) of target compounds, thus, SPME sampling conditions can be adjusted to favor a selected group of compounds, such as organic acids in tobacco.

Keywords: Optimization; Headspace sampling; Tobacco; Solid-phase microextraction; Volatile compounds

1. Introduction

Since solid-phase microextraction (SPME) was introduced in 1989 [1], it has been increasingly accepted as a sensitive sampling technique for volatile organic analysis in environmental [2,3] and food [4,5] industries among others [6,7]. The key configuration of SPME device is a silica fiber coated with a polymer phase (e.g. polydimethylsiloxane or carbowax). Adsorption of target compounds onto the polymer coated silica fiber can be done either from the headspace of solid or liquid sample or by directly dipping the fiber into an aqueous solution using a variety of mixing techniques [8]. The loaded fiber is then inserted into the GC injection port at an elevated temperature, in which the absorbed volatile compounds are thermally desorbed into the head of a capillary column for gas chromatographic analysis with various detectors.

The sampling mechanism has made SPME a concentration tool for trace analysis. In some cases, its solvent-free nature has proved to be unique and beneficial. For example, it happened so often that early eluted peaks of interested were masked by the huge solvent peak in the practice of fast GC analysis, particularly under low split ratio or splitless mode for a greater sensitivity. This masking problem did not exist when a solvent-free injection technique such as SPME was employed [9]. Another frequently used technique for the combined technique of SPME and fast GC was the use of cryotrapping followed by ballistic thermal desorption to improve the refocusing effect and the resolution of early eluted peaks. Usually, a mini-cryotrap was installed at the initial section of the capillary column for this purpose [4,10,11]. The solvent-free condition of SPME made multiple injections an easy task in the above cryo-

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